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RADIOCARBON DATING OF THE HUMAN OCCUPATION OF AUSTRALIA PRIOR TO 40 ka BP—SUCSESSES AND PITFALLS

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ABSTRACT. Charcoal samples from ancient human occupation sites in Australia have been subjected to a rigorous pretreatment and stepped combustion regime in order to explore the possibility that these sites may be older than previous radiocarbon dating had suggested. In one case, the Devil's Lair site in southwest Australia, the methodology has clearly removed vestiges of contamination by more modern carbon and has led to a revised radiocarbon chronology that provides evidence for human occupation of southwest Australia by at least 44 ka BP and probably by 46–47 ka BP. In contrast, charcoal from the Nauwalabila site has been so severely altered that insufficient of the original carbon remains for reliable ¹⁴C dating. Finally, where the charcoal is well preserved, such as at the Carpenter's Gap site, the new results provide reassurance that earlier ¹⁴C results of ~40 ka BP are indeed true ages and are not simply at the limit of the ¹⁴C technique.

INTRODUCTION

The upper limit to radiocarbon ages of the human occupation of Australia has been stubbornly stuck at ~40 ka for many years. This has led some authors to assert that humans arrived in Australia at this time (Allen and Holdaway 1995; O'Connell and Allen 1998). Recent evidence from other techniques, most notably thermoluminescence (TL) and optically stimulated luminescence (OSL), points however, to a more ancient arrival (Roberts et al. 1990, 1994). Is the 40 ka limit real, in which case the luminescence dating is suspect? Or is the luminescence dating correct, and 40 ka represents a "radiocarbon barrier" imposed by the inability to remove traces of younger carbon from samples at the level of <1% modern carbon?

In order to address this important question of the antiquity of human occupation of the Australian continent, a rigorous pretreatment and combustion methodology (acid-base-wet oxidation followed by stepped combustion—ABOX-SC) has been developed at the Australian National University (ANU) to remove as much as possible of non-original carbon from ancient charcoal (Bird et al. 1999). The application of this technique to three "case studies" of important sites in Australian archaeology is presented here.

METHODS

Details of the ABOX-SC technique are given by Bird et al. (1999). Briefly, charcoal that has been hand-picked from a particular archaeological level, or concentrated by sieving and flotation is subjected to the following pretreatment:

1. Treat with 6M HCl
2. Treat with 1M NaOH
3. Treat with H₂SO₄/K₂Cr₂O₇ (0.1M/2M) solution at 60 °C for up to 14 hr.

The material that remains after this treatment is then placed in a silica tube with CuO and silver wire, and subjected to a three-stage combustion procedure. The first stage, at 340 °C, is performed in the

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presence of O₂ gas; the CO₂ evolved during this step is usually discarded. The second and third steps are at 650 and ~900 °C, and utilize the oxygen from decomposition of the CuO. Carbon dioxide from each of these two steps is collected and graphitized separately using catalytic reduction on iron at 650 °C in the presence of H₂ gas.

The ¹⁴C/¹³C ratios of the samples were measured by accelerator mass spectrometry on the 14UD accelerator at the ANU. A fractionation correction was applied to the measured ratio assuming a value of −25‰ for the δ(¹³C) of the charcoal. Any uncertainty resulting from this assumption will be small compared to the statistical uncertainties on the results from these old samples.

THE SITES AND RESULTS

The Devil's Lair Site: Human Occupation Dated by Radiocarbon to Pre-40 ka BP

Devil's Lair is a limestone cave in the Margaret River region of West Australia. The name has been adopted by a nearby vineyard that makes excellent (and expensive) red wines. The stratigraphic sequence in the cave consists of 660 cm of sandy sediments intercalated with flowstone and bands of carbonate-cemented sediment. There is evidence for intermittent human occupation of the cave down to layer 30 at a depth of ~350 cm, with hearths, bone, and stone artifacts found throughout. Layer 30 represents a fan of topsoil that accumulated rapidly following widening of the cave mouth, and contains the earliest evidence for occupation of the cave itself. Below layer 30, in-situ artifacts are represented by only three stone flakes in level 34 and a single small flake in layer 37. These were presumably washed into the cave with the sediments and indicate human activity in the surrounding area. The site has been described in detail by Dortch (1979a, 1979b). Earlier radiometric dating of the site using untreated or alkali-washed charcoal produced a coherent chronology down to ~3m, but below 325 cm the ages were highly variable, and the oldest date was 38 ka (Dortch and Dortch 1996). Accelerator mass spectrometry (AMS) dates on charcoal subjected to a more rigorous acid-base-acid pretreatment do not show the variability, but appear to reach a plateau at ~40 ka below 325 cm (Turney et al. 2000).

Charcoal from a series of depths in the sequence below 250 cm has been subjected to the ABOX-SC treatment. Results are superimposed on a diagram of the sedimentary sequence in Figure 1. Occupation of the cave is dated to around 44 ka BP, and level 37, which contains the deepest artifact, is dated to around 45 ka BP. Even older ages are obtained for the sterile layers below level 37, which indicates that the dates above this level are likely to be real ages, and are not simply reflecting the limit of the technique. A more detailed report of this work is forthcoming (Turney et al. 2000). For two of the samples, a variety of other common pretreatments were also undertaken in order to determine whether a simpler pretreatment than ABOX-SC might give equivalent results. Table 1 shows a comparison between the different pretreatments. Clearly, the full ABOX-SC treatment is required if reliable ages are to be obtained for the >40 ka BP samples from this site.

Calibration of the ¹⁴C time scale in terms of calendar years is problematic in the time range beyond 40 ka. Simply taking into account the difference between the Libby half-life of 5568 years (used to calculate the ¹⁴C age) and the presently accepted value of 5730 yr, however, increases a 45 ka ¹⁴C age by 1300 yr. Recently, two calibrations that extend back beyond 40 ka BP have been reported (Kitagawa and van der Plicht 1998; Voelker et al. 1998), and indicate offsets between sidereal and ¹⁴C ages of 1–2 ka at 45 ka BP. Hence, the new ¹⁴C results reported here imply the presence of people in the Devil's Lair area by ~46,000–47,000 years ago. OSL, ESR and U-series ages are also available for this site (Turney et al. 2000), and the revised ¹⁴C chronology from this work is in good agreement with them beyond 40 ka BP.

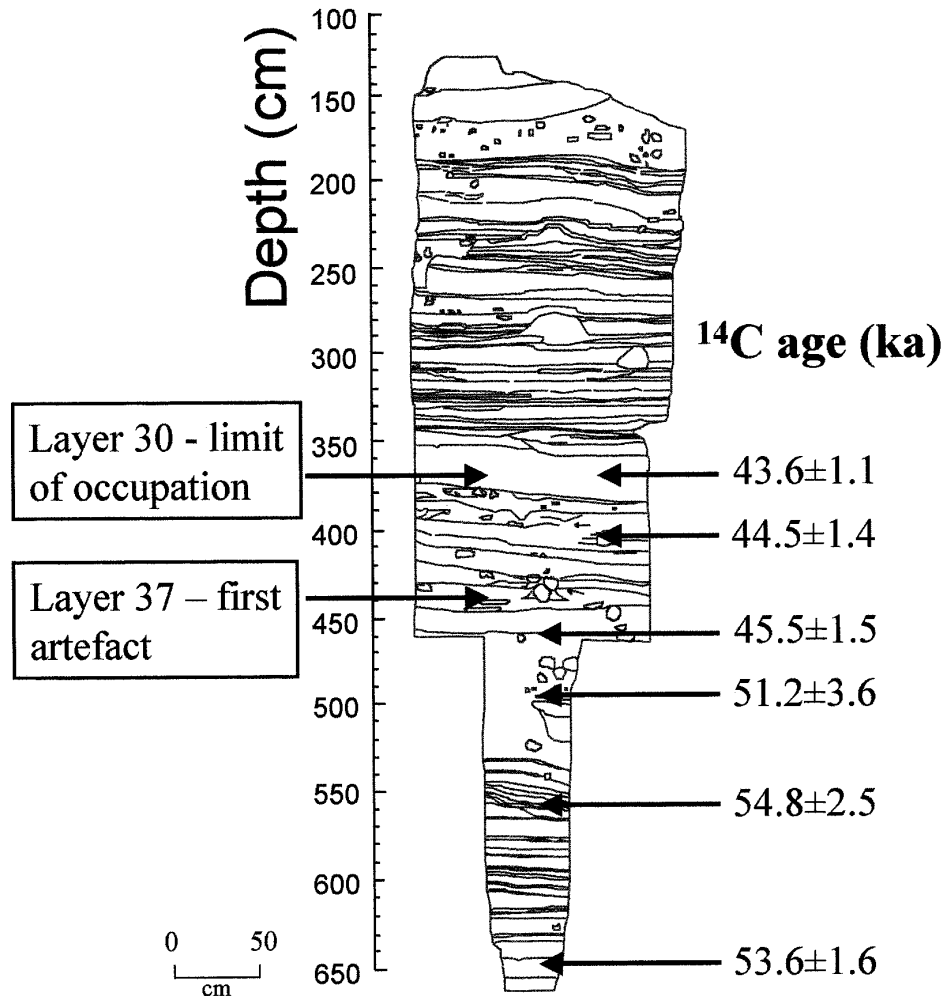


Figure 1 A schematic cross-section of the Devil's Lair excavation showing the significant markers in the sequence and ABOX-SC-AMS radiocarbon ages

These results constitute the first definitive evidence from ^{14}C dating for the presence of humans in Australia before 40 ka BP, and indicate that people had reached this extreme southwestern tip of the continent by at least 46–47 ka ago. Crucial to this conclusion has been the combination of a stringent pre-treatment regime and a stepped combustion procedure. Between them, these seem to eliminate substantially more of the carbon incorporated into the samples during their period of burial than do more conventional pre-treatments followed by bulk combustion.

The Nauwalabila Site: Failure of Radiocarbon Dating

Nauwalabila is a rock overhang in the Kakadu National Park area of the Northern Territory. It comprises a 3-m-deep sequence of sands containing abundant evidence of human occupation. The site came to prominence following claims, based on thermoluminescence (TL) dating, that it had first been occupied at some time between 60 and 53 ka BP. Previous ^{14}C dating of this site had, however, failed to yield ages older than 30 ka BP, casting some doubt over the TL results.

Table 1 Comparison of various pretreatments for samples from the Devil's Lair site

Layer/depth (cm)	Pretreatment	Combustion fraction (°C)	Lab nr (ANUA-)	Radiocarbon age (ka BP) ^a
30/365	HCl wash only	880 (i) ^b	11506	42.0 ± 1.6
30/365	HCl wash only	880 (ii) ^b	11505	41.1 ± 1.9
30/365	ABOX	340	11504	41.4 ± 1.2
30/365	ABOX	650	11503	42.6 ± 1.9
30/365	ABOX	880	11502	43.6 ± 1.0
39/465	Base wash only	340	10302	34.0 ± 0.5
39/465	Base wash only	880	10303	40.0 ± 0.8
39/465	AOX	880	10304	45.4 ± 1.2
39/465	ABOX	340	10301	41.4 ± 1.3
39/465	ABOX	880	11511	45.5 ± 1.5

^aRaw radiocarbon ages—no blank or $\delta^{13}\text{C}$ corrections have been applied.

^b CO_2 evolved during first and second half of combustion at 880 °C was collected and graphitized separately.

In the present work, charcoal from the problematic depth range between 1.5 and 3 m, and from different size fractions, has been subjected to the ABOX-SC treatment. Figure 2 summarizes the results from both this and earlier work. A more complete account of this work, including a catalogue of all of the data summarized in Figure 2, will be published elsewhere (Bird et al. forthcoming). Down to ~1.3 m, the ages from radiometric ^{14}C dating are in stratigraphic order and extend to ~15 cal ka. In addition, there is good agreement between the radiocarbon chronology and a TL date at ~1.1 m. Below 1.3 m, however, the radiometric ^{14}C ages become highly variable. The TL dates, on the other hand, continue to increase monotonically with depth. It is clear from Figure 2 that the ABOX-SC treatment does nothing to reduce the variability in ^{14}C ages below 1.3 m, nor does it produce any older dates than had been obtained previously by radiometric counting. Further, there is a lack of any clear correlation between age and size-fraction.

The onset of variability of the ^{14}C ages coincides with a dramatic fall in the amount of charcoal in the sequence, and the appearance of pisoliths. The latter are iron oxide nodules which are indicative of the presence of a fluctuating water table at some time in the past. It appears that, below 1.3 m, much of the charcoal has disappeared. Further, what remains has been profoundly altered. Carbon contents of the charcoal were as low as 4% and iron and silica contents as measured by energy-dispersive X-ray analysis were high. The processes that led to this degradation were presumably also responsible for the disappearance of much of the charcoal from this part of the sequence. Several of the youngest ages cluster around 8–9 ka, and these may indicate the time at which the water table was high. A mechanism for degradation of the charcoal has not yet been identified, although microbial action is suspected. Further work is planned to elucidate these processes.

The conclusion in this case is that, below a depth of 1.3 m, the ^{14}C ages are simply wrong. The moral is that there is some material that is so profoundly altered and contains so little of the original carbon that no amount of pretreatment can save it. Carbon analysis and electron microscopy of suspect material can save time spent on futile time-consuming pretreatments.

For this Nauwalabila site, therefore, the ^{14}C results are mute concerning the question of the antiquity of human occupation of the site. There thus appears to be no good reason to doubt the TL ages, and indeed, support for their validity is provided by the good agreement between ^{14}C and TL ages at 1.1 m. They are also lent credence by the Devil's Lair results reported above which firmly establish human occupation of the Australian continent by ~46–47 ka BP.

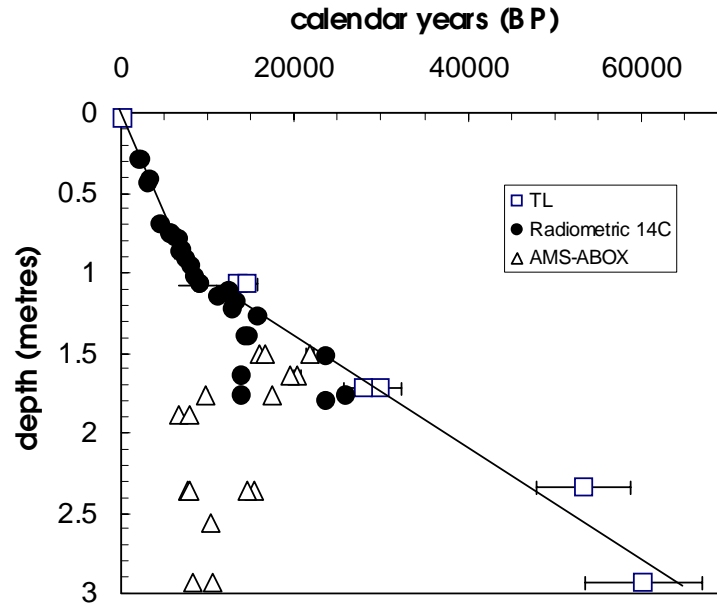


Figure 2 Comparison between radiometric ^{14}C dates, AMS ^{14}C dates on ABOX-SC treated charcoal, and TL dates for the Nauwalabila site

Table 2 Results of radiocarbon measurements for ancient Australian archaeological sites

Site and sample description	ANUA nr	Radiocarbon age (ka BP) ^a
<i>Carpenter's Gap</i>		
CG1-A45, 90 cm, ABOX 880 ^b	8130	33.2 ± 0.6
CG1-A45, 90 cm, ABOX 660 ^b	8227	33.0 ± 0.7
CG1-A45, 90 cm, ABOX 340 ^c	8226	33.1 ± 0.8
CG1-A45, 90 cm, ABOX 660 ^c	8222	32.8 ± 0.5
CG1-A45, 90 cm, ABOX 880 ^c	7626	33.6 ± 0.5
CG1-A48, 96 cm, ABOX 340	7615	39.8 ± 0.6
CG1-A48, 96 cm, ABOX 660	7616	40.6 ± 0.8
CG1-A48, 96 cm, ABOX 880	7617	40.3 ± 0.8
<i>Riwi</i>		
Riwi 13, 65 cm, ABOX 920	13005	41.3 ± 1.0
Riwi 16, 95 cm, ABOX 920	13006	40.7 ± 1.3
<i>Cuddie Springs</i>		
CS1/1, 1.05–1.15 m, ABOX 880 ^b	10011	28.74 ± 0.34
CS1/3, 1.05–1.15 m, ABOX 920 ^c	13012	28.59 ± 0.48
CS3/1, 1.15–1.35 m, ABOX 880	10319	32.00 ± 0.55
CS2/1, 1.35–1.55 m, ABOX 880 ^b	10012	28.78 ± 0.35
CS2/2, 1.35–1.55 m, ABOX 880 ^c	12309	31.34 ± 1.00

^a A blank correction of 0.10 ± 0.02 pMC has been subtracted and a $\delta^{13}\text{C}$ value of -25‰ for charcoal has been assumed

^{b,c} Independent preparations from two pieces of charcoal from the same level

The Carpenter's Gap Site: Reliable Confirmation of Previous Radiocarbon Ages at ~40 ka BP

Carpenter's Gap is a rock shelter site in northwestern West Australia. The sequence is 1 m deep, and artifacts are found down to the bedrock base (O'Connor 1995). Earlier ^{14}C dating indicated ages of ~40 ka at the base of the sequence (McConnell and O'Connor 2000). In contrast to the Nauwalabila site, there is abundant charcoal all the way down the sequence, and its state of preservation is remarkable.

Multiple samples of charcoal from three levels near the base of the sequence have been ABOX-SC treated and measured by AMS. The results are presented in Table 2 above. Clearly, the ages for a given level are all consistent, both internally and with earlier ^{14}C measurements, and establish beyond reasonable doubt that this site was first occupied by humans 40,000 ^{14}C years ago. Even the low-temperature fractions of the stepped combustion procedure yield an age similar to the high-temperature fractions, indicating that residual contamination after the chemical pretreatment is minimal. The message from this site is that if one starts with good charcoal, then the dates are likely to be reliable, even at 40 ka BP.

Selected samples from two other sites that had earlier been ^{14}C dated to ~30 ka or more using conventional pretreatments and combustion procedures were also subjected to the full ABOX-SC procedure and ^{14}C dated by AMS in the course of this work in order to determine whether or not they might be significantly older. Their locations are shown in Figure 3 and the results are presented in Table 2. Riwi is a limestone cave in the southern Kimberleys with a 1-m-deep sequence of human occupation. Details may be found in Balme (2000). Of the two samples, Riwi 13 is from a hearth at a depth of 65 cm, and its age is very similar to that at the base of the sequence at Carpenter's Gap, which is 200 km to the northwest. It provides additional evidence that humans were active in the Kimberley region of northwest Australia by at least 40 ka BP. Riwi 16 is an isolated charcoal fragment that was picked from near the base of the sequence; its age would suggest that it may be out of sequence, as discussed by Balme (2000). Cuddie Springs is an ephemeral lake site on the other side of the continent in north central NSW. It is of considerable interest because human artefacts and bones of extinct megafauna are found in the same archaeological layers, suggesting strongly that humans and megafauna coexisted at this site (Field and Dodson 1999). Previous AMS ^{14}C dating of charcoal from these layers, using conventional ABA pretreatment, ranged from 28.8 to 33.7 ka BP, and the results obtained here confirm these earlier results.

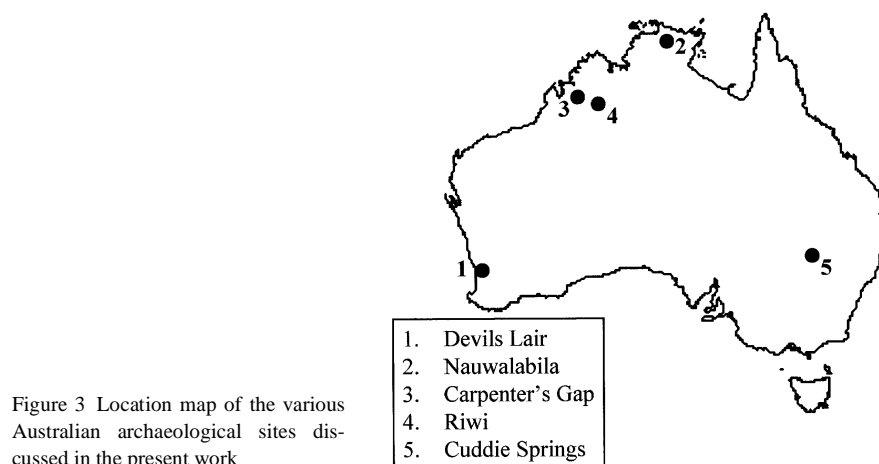


Figure 3 Location map of the various Australian archaeological sites discussed in the present work

CONCLUSIONS

Charcoal samples from ancient human occupation sites in Australia have been subjected to a rigorous pretreatment and stepped combustion regime in order to explore the possibility that these sites may be older than earlier ^{14}C dating had suggested. In one case, the Devil's Lair site in southwest Australia, the ABOX-SC methodology has clearly removed vestiges of contamination by more modern carbon and has led to a revised ^{14}C chronology which provides evidence for human occupation of southwest Australia by at least 45 ka BP, and probably 46–47 ka. In marked contrast, the charcoal from depths below 1.3 m at the Nauwalabila site has been found to have been so severely altered during an episode when the water table was high that not even the ABOX-SC methodology can recover the original carbon. Finally, where the charcoal is well preserved, such as at the Carpenter's Gap site, the ABOX-SC results provide reassurance that earlier ^{14}C results of ~40 ka BP are indeed true ages and are not simply at the limit of the radiocarbon technique.

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